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IS 260 (2001): Aluminium sulphate, non-ferric [CHD 1: Inorganic Chemicals]

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(दूसरा पुनरीक्षण)

Indian Standard

ALUMINIUM SULPHATE, NON-FERRIC — SPECIFICATION

(Second Revision)

ICS 71.060.50

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

June 2001

Price Group 5

General Inorganic Chemicals Sectional Committee, CHD 3

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the General Inorganic Chemicals Sectional Committee had been approved by the Chemical Division Council.

This standard was originally published in 1950 covering only the technical grade of aluminium sulphate, non-ferric and subsequently revised in 1969, incorporating pure analytical reagent grades of the material and to keep abreast with the progress made by the industry in this field. Flame photometric method was prescribed for the determination of sodium and potassium. Since widely different types of instruments are available for this determination, detailed instructions have not been given.

In this present (second) revision, EDTA method for determination of aluminium has been modified. The specified limit for arsenic has also been suitably modified for all the three grades.

Aluminium sulphate, non-ferric, is a purer form of aluminium sulphate than alumino-ferric. It is used as a mordant in the dyeing of textiles, as a loading material for better grades of paper, for clarifying oils and fats, in the preparation of aluminium resinates and other pure aluminium salts. The pure grade of the material is also used for the pharmaceutical preparations.

Aluminium sulphate, non-ferric, is also used in large quantities for purification of water used for injection into oil-bearing formations for pressure maintenance.

Composition of the Committee responsible for its formulation is given in Annex M.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard
ALUMINIUM SULPHATE, NON-FERRIC — SPECIFICATION
(Second Revision)

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for aluminium sulphate, non- ferric.

2 REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
264 : 1976	Nitric acid (*second revision*)
265 : 1993	Hydrochloric acid (*fourth revision*)
321 : 1964	Absolute alcohol (*revised*)
323 : 1959	Rectified spirit (*revised*)
1070 : 1992	Reagent grade water (*third revision*)
2088 : 1983	Methods for determination of arsenic (*second revision*)
4905 : 1968	Methods for random sampling

3 GRADES

The material shall be of the following three grades:

a) *Technical* (TECH) — intended for use as a mordant in the dyeing of textiles and as a loading material for better grades of paper;

b) *Pure* — intended for use in pharmaceutical preparations and where pure grade of the material is required, namely, for clarifying oils and fats, preparation of aluminium resinates and other pure aluminium salts; and

c) *Analytical Reagent* (AR) — for use as a reagent in chemical analysis.

4 REQUIREMENTS

4.1 Description

The material shall be in the form of white, slightly deliquescent crystals or granules.

4.2 The material shall also comply with the requirements given in Table 1 when tested in accordance with the methods prescribed in Annexes A to K. Reference to the relevant Annex is given in col 6 of Table 1.

5 PACKING AND MARKING

5.1 Packing

The material shall be supplied in bulk or in packages as agreed to between the purchaser and the supplier.

5.2 Marking

5.2.1 The packages shall be securely closed and marked with the following information:

a) Name and grade of the material,

b) Mass of the material,

c) Indication of the source of manufacture,

d) Month and year of manufacture, and

e) Lot number in code or otherwise to enable the batch of manufacture to be traced from records.

5.2.2 *BIS Certification Marking*

The packages may also be marked with the Standard Mark.

5.2.2.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5.2.3 In case of analytical reagent grade, the analysis of the material, details of analysis of the material in respect of the characteristics laid down in col 5 of Table 1 shall also appear on the label.

6 SAMPLING

The method of drawing representative samples of the material, number of tests to be performed and the criteria for determining the conformity of the material to the requirements of this specification shall be as prescribed in Annex L.

7 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and reagent grade water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities, which affect the results of analysis.

Table 1 Requirements for Aluminium Sulphate, Non-Ferric

(*Clause* 4.2)

Sl No.	Characteristic	Requirement			Method of Test (Ref to Annex)
		TECH Grade	Pure Grade	AR Grade	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Insoluble matter, percent by mass, *Max*	0.30	0.02	0.005	A
ii)	*p*H (of 5 percent solution), *Min*	2.8	2.9	2.2	B
iii)	Heavy metals (as Pb), parts per million, *Max*	—	40	10	C
iv)	Iron (as Fe), percent by mass, *Max*	0.01	0.004	0.002	D
v)	Chlorides	—	—	To pass test	E
vi)	Arsenic (as As_2O_3), parts per million, *Max*	—	8	5	F
vii)	Ammonium salts (as NH_3), percent by mass, *Max*	—	0.025	0.025	G
viii)	Aluminium (as Al_2O_3), percent by mass	17 (*min*)	16.6-17.6	16.6- 17.6	H
ix)	Sodium (as Na), percent by mass, *Max*	—	—	0.05	J
x)	Potassium (as K) percent by mass, *Max*	—	—	0.01	K

ANNEX A

[*Table* 1, *Sl No.* (i)]

DETERMINATION OF INSOLUBLE MATTER

A-1 REAGENTS

A-1.1 Dilute Sulphuric Acid — Approximately 1 N.

A-2 PROCEDURE

A-2.1 Preparation of Test Sample

Crush about 200 g of the material to pass 1.00 mm IS Sieve. Keep this prepared sample in a clean glass-stoppered weighing bottle.

A-2.2 Weigh accurately about 10 g of the prepared sample mix with 10 ml of dilute sulphuric acid, and dissolve in 100 ml of water by stirring and warming, if necessary. Filter through a tared filter paper or a sintered glass crucible (G 4); wash the residue till it is free from soluble compounds, and dry to constant weight at 105 to 110°C.

A-3 CALCULATION

$$\text{Insoluble matter, percent by mass} = \frac{m}{M} \times 100$$

where

m = mass in g of the residue, and

M = mass in g of the prepared sample taken for the test.

ANNEX B

[*Table* 1, *Sl No.* (ii)]

DETERMINATION OF *p*H

B-1 PROCEDURE

Take 5 g of the material and dissolve in 100 ml of freshly boiled and cooled water. Determine *p*H of the solution by means of a suitable *p*H meter, using glass electrodes.

ANNEX C

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF HEAVY METALS

C-1 OUTLINE OF THE METHOD

Heavy metals are determined by comparing the colour produced by the material with sodium sulphide solution against that produced by standard lead solution.

C-2 APPARATUS

C-2.1 Nessler Cylinders — 50 ml capacity.

C-3 REAGENTS

C-3.1 Dilute Ammonium Hydroxide — Approximately 4 N.

C-3.2 Sodium Sulphide Solution — 10 percent (*m*/*v*).

C-3.3 Concentrated Nitric Acid — *See* IS 264.

C-3.4 Standard Lead Solution

Dissolve 1.60 g of lead nitrate in water, add 1 ml of concentrated nitric acid and dilute the solution to 1 000 ml. Pipette out 10 ml of this solution and then dilute to 1 000 ml. One millilitre of the final solution is equivalent to 0.01 mg of lead (as Pb). The diluted solution shall be freshly prepared.

C-4 PROCEDURE

Weigh accurately 1.0 g of the prepared sample and dissolve it in about 20 ml of water. Transfer the solution to a Nessler cylinder; add 4 drops of sodium sulphide solution. Carry out a control test in another Nessler cylinder using 4 ml of standard lead solution in place of the sample in case of pure grade and one millilitre in case of analytical reagent grade, and the same quantities of other reagents in the same total volume of reaction mixture. Compare the colour produced in the two cylinders.

C-4.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced in the test with the sample is not greater than that produced in the control test.

ANNEX D

(*Table* 1, *Sl No.* (iv))

DETERMINATION OF IRON

D-1 OUTLINE OF THE METHOD

Iron is determined colorimetrically by visual comparison using butanolic potassium thiocyanate solution.

D-2 APPARATUS

D-2.1 Nessler Cylinders — 50 ml capacity.

D-3 REAGENTS

D-3.1 Concentrated Hydrochloric Acid — *See* IS 265.

D-3.2 Ammonium Persulphate — Solid.

D-3.3 Butanolic Potassium Thiocyanate Solution

Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient *n*-butanol to make up to 100 ml and shake vigorously until the solution is clear.

D-3.4 Standard Iron Solution

Dissolve 0.702 g of ferrous ammonium sulphate [$Fe(NH_4)_2(SO_4)_2.6H_2O$] in 10 ml of dilute sulphuric acid (10 percent *v/v*) and dilute with water to 1 000 ml. Take 10 ml of this solution and dilute to 100 ml. One millilitre of this solution contains 0.01 mg of iron (as Fe).

D-4 PROCEDURE

Dissolve 10.0 g of the prepared sample in water and make up the volume to 100 ml. Transfer 10 ml of this solution into a Nessler cylinder and dilute with 30 ml of water. Add 1 ml of concentrated hydrochloric acid, 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution. Shake vigorously for 30 seconds and allow the butanol layer to separate. Carry out a control test in the second Nessler cylinder using the following amounts of standard iron solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture:

a) 10 ml in case of technical grade,
b) 4 ml in case of pure grade, and
c) 2 ml in case of analytical reagent grade.

D-4.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the colour produced in the test with the material is not greater than that produced in the control test.

ANNEX E

[*Table* 1, *Sl No.* (v)]

TEST FOR CHLORIDES

E-1 REAGENTS

E-1.1 Dilute Nitric Acid — 5 N approximately.

E-1.2 Silver Nitrate Solution — 5 percent (*m/v*).

E-2 PROCEDURE

Dissolve 1.0 g of the prepared sample in 50 ml of water. Add one millilitre of dilute nitric acid and one millilitre of silver nitrate solution. The material shall be considered to have satisfied the requirements of the test if no opalescence is produced.

ANNEX F

(*Table* 1, *Sl No.* (vi))

DETERMINATION OF ARSENIC

F-1 PROCEDURE

Dissolve 1.0 g of the prepared sample in 10 ml of water. Carry out the test for arsenic as prescribed in IS 2088, using for comparison a stain obtained with 0.006 mg of arsenic as As_2O_3 in case of pure grade, 0.004 mg in case of technical grade and 0.002 mg in case of analytical reagent grade of the material.

F-1.1 The material shall be considered to have satisfied the requirements of the test if the length as well as the intensity of the stain obtained with the sample is not greater than that produced in the control test.

ANNEX G

[*Table* 1, *Sl No.* (vii)]

DETERMINATION OF AMMONIUM SALTS

G-1 OUTLINE OF THE METHOD

Ammonium salts are determined with Nessler solution, which reacts with ammonia to form a reddish-brown colloidal suspension.

G-2 APPARATUS

G-2.1 Nessler Cylinders — 50 ml capacity.

G-3 REAGENTS

G-3.1 Sodium Hydroxide Solution — Approximately 5 N.

G-3.2 Nessler Solution

Dissolve 10 g of potassium iodide in 10 ml of ammonia-free water, and add to it slowly with stirring saturated mercuric chloride solution until a slight permanent precipitate forms. Add 30 g of potassium hydroxide and when it has dissolved, add 1 ml more of mercuric chloride solution, and dilute to 200 ml with ammonia-free water. Allow settling overnight, decanting the clear solution and keeping it in a bottle closed with a well-fitting rubber stopper.

G-3.3 Standard Ammonium Chloride Solution

Dissolve 0.314 1 g of ammonium chloride in water and make up to 1 000 ml. One millilitre of this solution is equivalent to 0.1 mg of ammonia (as NH_3).

G-4 PROCEDURE

Weigh accurately 1.0 g of the prepared sample and dissolve in 20 ml of water in a Nessler cylinder. Add 5 ml of sodium hydroxide solution and one millilitre of Nessler solution. Make up the solution to 50 ml. Carry out a control test in another Nessler cylinder with 2.5 ml of standard ammonium chloride solution in place of the material and the same quantities of other reagents. Compare the colour produced in the two cylinders.

G-4.1 The limit prescribed shall be taken as not having been exceeded if the intensity of colour produced in the test with the material is not greater than that produced in the control test.

ANNEX H

[*Table* 1, *Sl No.* (viii)]

DETERMINATION OF ALUMINIUM

H-1 GENERAL

Two methods are prescribed, namely (a) gravimetric method, and (b) titration with EDTA solution. Gravimetric method shall be used, as referee method in case of dispute.

H-2 METHOD A (GRAVIMETRIC METHOD)

H-2.1 Reagents

H-2.1.1 *Ammonium Chloride Solid*

H-2.1.2 *Concentrated Nitric Acid* — *See* IS 264.

H-2.1.3 *Dilute Ammonium Hydroxide* — Approximately 4 N.

H-2.1.4 *Methyl Red Indicator*

Dissolve 0.1 g of methyl red in 100 ml of rectified spirit (*see* IS 323).

H-2.1.5 *Ammonium Nitrate Solution* — Approximately 2 percent.

H-2.2 Procedure

Weigh accurately about 1 g of the prepared sample and dissolve it in 100 ml of water. Filter to remove the undissolved matter and wash the filter paper thoroughly. Add 5 g of ammonium chloride and 2 drops of concentrated nitric acid to the filtrate and washings; heat to boiling and add dilute ammonium hydroxide, drop by drop, until the solution smells faintly of ammonia and shows distinct yellow colour on the addition of two drops of methyl red indicator. Boil for 2 min, filter immediately through filter paper (Whatman No. 40 or equivalent) and wash the residue with hot ammonium nitrate solution till the filtrate is free from chlorides. Dry the residue and ignite to constant weight at 1 100 to 1 200°C in tared porcelain or a silica crucible.

H-2.3 Calculation

Aluminium (as Al_2O_3), percent by mass =

$$100 \times \frac{M}{M_1} - A \times 1.429\,7$$

where

M = mass, in g, of the ignited residue;

A = percent by mass of iron as determined in Annex D; and

M_1 = mass in g of the prepared sample taken for the test.

H-3 METHOD B (EDTA METHOD)

H-3.1 Reagents

H-3.1.1 *Disodium Ethylenediamine Tetra Acetate (EDTA) Solution* — 0.05M.

Dissolve 18.612 5 g of disodium ethylenediamine tetra acetate dihydrate in water and make the volume to one litre in a volumetric flask.

H-3.1.2 *Concentrated Sulphuric Acid*

H-3.1.3 *Hexamine (Hexamethylene Tetramine) Analar*

H-3.1.4 *Xylenol Orange Indicator*

Prepare 0.5 percent in water and add 1 to 2 drops of dilute sodium hydroxide solution.

H-3.1.5 *Standard Lead Nitrate Solution* — 0.05 N.

H-3.2 Procedure

Dissolve 5 g of prepared sample in water. Acidify it by adding 1 to 2 ml of concentrated sulphuric acid and make up the volume to 500 ml in a volumetric flask. Take an aliquot of 50 ml into a conical flask. Add 50 ml of 0.05 M EDTA solution and heat to just boiling. Cool it and add hexamine powder to adjust the *p*H to 5. Add few drops of xylenol orange indicator. Titrate the excess EDTA solution with standard lead nitrate solution until the colour changes from yellow to pink.

H-3.3 Calculation

Calculate the aluminium content of the material on the assumption that one millilitre of 0.05 M EDTA solution is equivalent to 0.002 55 g of aluminium (as Al_2O_3).

ANNEX J

[*Table* 1, *Sl No.* (ix)]

DETERMINATION OF SODIUM

J-1 PROCEDURE

Determine sodium by flame photometer at 589 nm according to the directions of the manufacturer of the apparatus (*see* Foreword).

ANNEX K

[*Table* 1, *Sl No.* (x)]

DETERMINATION OF POTASSIUM

K-1 PROCEDURE

Determine potassium by flame photometer at 766.5 nm according to the directions of the manufacturer of the apparatus (*see* Foreword).

ANNEX L

(*Clause* 6.1)

SAMPLING OF ALUMINIUM SULPHATE, NON-FERRIC

L-1 GENERAL REQUIREMENTS OF SAMPLING

In drawing samples the following precautions and directions shall be observed.

L-1.1 Samples shall not be taken in an exposed place.

L-1.2 The sampling implement shall be clean and dry when used.

L-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling implement and the containers for samples from adventitious contaminations.

L-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by suitable means.

L-1.5 The samples shall be placed in suitable, clean, dry and air-tight glass containers which shall be sealed air-tight after filling and marked with full details of sampling.

L-2 SCALE OF SAMPLING

L-2.1 Lot

In a single consignment of the material all the containers of the same size and containing the same grade of material and drawn from the same batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture or of different types or sizes of containers, those belonging to the same batch, type and size shall be grouped together and each such group shall constitute a separate lot.

L-2.1.1 For ascertaining the conformity of the material in a lot to the requirements of the specification, tests shall be carried out for each lot separately. The number of containers to be selected at random from lots of different sizes shall be in accordance with Table 2.

L-2.1.2 Random number tables shall be used for selecting the containers. In case such tables are not available, the following procedure is recommended for use:

Starting from any container at random, count them in one order as 1,2,3...etc, up to *r* and so on where *r* is the integral part of N/n (N and n being the lot size and sample size respectively). Every *r*th container thus counted shall be withdrawn to constitute the sample.

Table 2 Scale of Sampling

(*Clause* L-2.1.1)

Lot Size (N)	Sample Size (n)
(1)	(2)
Up to 25	3
26 to 50	4
51 to 100	5
101 and above	7

L-3 PREPARATION OF TEST SAMPLES

L-3.1 From each of the containers selected, draw a small representative portion of aluminium sulphate approximately 100 g in weight.

L-3.2 Out of these portions, a small but equal quantity of the material shall be taken and mixed thoroughly to form a composite sample of weight about 200 g. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

L-3.3 The referee sample shall bear the seal of both the purchaser and the supplier and shall be kept at a place agreed to between them, to be used in case of dispute between the two.

L-4 NUMBER OF TESTS

Tests for the determination of the entire characteristic given in Table 1 shall be conducted on the composite sample.

L-5 CRITERION FOR CONFORMITY

For declaring the conformity of the lot to the requirements of all the characteristics tested on the composite sample, the test results shall meet the corresponding requirements specified in Table 1.

ANNEX M

(*Foreword*)

COMMITTEE COMPOSITION

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards : Monthly Additions'.

This Indian Standard has been developed from Doc : No. CHD 3 (792).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110 002
Telephones : 323 01 31, 323 33 75, 323 94 02
Telegrams: Manaksanstha
(Common to all offices)

Regional Offices :	Telephone
Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110 002	323 76 17 323 38 41
Eastern : 1/14 C. I. T. Scheme VII M, V. I. P. Road, Kankurgachi CALCUTTA 700 054	337 84 99, 337 85 61 337 86 26, 337 91 20
Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160 022	60 38 43 60 20 25
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